



April 4, 2006

Technical Division Note

TD-06-008

Technical Specifications for
High RRR Grade Niobium
Sheet and Rod
for use in
Superconducting Cavities

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(XXXX)

<u>Revision</u>	<u>ER / ECO</u>	<u>Date</u>	<u>Description</u>	<u>Approval(s)</u>

1.0 **GENERAL**

The Fermi National Accelerator Laboratory is involved in the design and construction of superconducting particle accelerators. Many components require dense, non alloyed - high purity, high RRR, annealed Niobium. They are fabricated from plate and rod stock by machining as well as “deep drawing” or other metal-forming processes and joined by electron-beam welding. All Nb ingots shall be produced by the electron beam vacuum-melting process. Sheets shall be in the fully re-crystallized state. Final annealing of Nb sheet shall be performed in vacuum of 2×10^{-5} Torr or better. The final product shall be processed in such a way that the final composition of the sheets (after the final annealing) meets this specification, intended to assure high purity, fine grain size material that is free from oxide inclusions, scale inclusions and internal laminations and fissures.

2.0 **APPLICABLE DOCUMENTS:**

2.1 *Applicability*

The following documents of the issue in effect on date of invitation for bids or request for bids or request for proposal form a part of this specification to the extent specified herein.

- ASTM B-391 Niobium and Niobium Ingot Alloys
- ASTM B-393 Standard Specification for Niobium and Niobium Alloy Strip, Sheet, Foil and Plate
- ASTM E-112 Standard method for determining the average grain size
- ASTM E-8 Method of tension testing of metallic materials.

2.2 *Source of Documents*

The documents may be obtained from the following sources:

ASTM Documents:

American Society of Testing and Materials

1916 Race Street

Philadelphia, PA 19103

3.0 **REQUIREMENTS**

The parent material shall conform to ASTM B391, “Niobium and Niobium Ingot Alloys”, R04200-Type 1, reactor grade unalloyed niobium with additional and overriding requirements as specified herein.

3.1 *Chemical Composition*

Each lot of annealed Nb sheet shall conform to the following requirements for chemical composition:

<u>Element</u>	<u>Max. Parts per Million (weight ppm)</u>
Ta	500
W	70
Ti	40
Fe	30
Si	30
Mo	50
Ni	30

All other metallic impurities should be < 30 ppm each. In addition, each niobium sheet (final product) shall conform to the following requirements for dissolved interstitial elements:

<u>Element</u>	<u>Max. Parts per Million (weight ppm)</u>
O	10
N	10
C	10
H	2

3.2 *Measurement of Chemical Composition*

The method of measurement of chemical composition shall be according to Section 5.1

4.0 **DESCRIPTION OF DELIVERED MATERIAL**

The delivered material shall conform to the following specifications:

4.1 *Electrical Requirements*

The final product shall have a measured Residual Resistivity Ratio (RRR) value greater than 300. This requirement overrides the chemical composition requirement in Section 3.0, since the combined contributions of the impurities as listed in the above table would produce an RRR value substantially below the required value. The method of measurement of the RRR value shall be specified in Section 5.2.

4.2 *Dimensions*

The dimensions of the delivered material shall be as specified in the purchase order and are net dimensions and forging scale shall be removed. The sheet surface flatness shall be according to ASTM B708: 2 % or better. The rod diameter should not vary by more than 4%. No oxide impregnation, subsurface inclusions of oxide or other foreign material will be acceptable.

4.3 *Metallurgical Requirements*

The final product shall be at least 95% recrystallized and exhibit uniform size and equi-axed grains that are predominantly ASTM #6 (0.045 mm) with no grains larger than ASTM #5 (0.064 mm) locally. The method of measurement shall be according to Section 5.3.

4.4 *Mechanical Properties*

The final product shall conform to the following mechanical properties at room temperature as per ASTM E-8:

- | | |
|--------------------------------|---|
| • Yield Strength (0.2% offset) | 7000 psi (48.2 N/mm ²) minimum
10200 psi (70.2 N/mm ²) maximum |
| • Tensile strength | 14000 psi (96.4 N/mm ²) minimum |
| • Elongation | 40 % minimum longitudinal |
| (1 inch gauge length) | 35 % minimum transverse |
| • Hardness, HV 10 | 60 maximum |

The yield strength and elongation in the longitudinal and transverse directions shall not differ by more than 10% of the respective value. For the tensile measurement, the rate-of-strain shall not exceed 0.010 inch/inch-minute through the 0.2% offset yield strength. The rate-of-strain may be increased to 0.05 inch/inch-minute thereafter.

The method of measurement shall be according to Section 5.3.

4.5 *Surface Finish*

The material shall be delivered in fully annealed condition with an “as etched” surface (free of oxide inclusions, cracks, blisters or laminations), with both surfaces having a clean “metallic” looking finish (metallic blank) and a surface roughness $R_t < 15 \mu\text{m}$. The delivered material shall be free of injurious internal or external imperfections of a nature that would interfere in the deep drawing operation of rf cavity components. Particular care should be taken in the final rolling and subsequent handling to avoid the inclusion and imbedding of particles of foreign material in the surface of the sheets. The method of measurement shall be according to Section 5.3.

5.0 **INSPECTION AND TESTING**

The tests listed below are the minimum required and are not intended to supplant any controls, examinations, inspections, or tests normally employed by the seller to assure the quality of the product. A minimum of two samples per sheet shall be taken from two sheets per each lot of Nb sheet covered by this specification. The specimens shall be prepared as per ASTM E 3. Samples should be sent to FNAL for additional measurements and acceptance.

5.1 *Chemical composition*

The method of measurement of chemical composition shall be by x-ray emission spectroscopy (ASTM E-902), spark source mass spectrometry (ASTM E-304) or other instruments with demonstrated equal sensitivity and accuracy, including dc plasma spectroscopy, optical emission spectroscopy or inductively coupled plasma spectroscopy.

5.2 *Residual Resistivity Ratio (RRR)*

RRR is defined as the ratio of D.C. bulk electrical resistivity at 293°K and at 4.2°K when the material is in a normal, non-superconducting state. Samples for RRR measurement shall be approximately 2.0mm x 5.0 mm x 50.0 mm in size. The high temperature measurement shall be made at 293 ± 1 K. The low temperature measurement may be made by one of two methods. It may be made above the critical temperature (9.2K), in which case the measurement temperature must be recorded. Alternatively, the low temperature resistance measurement may be made at 4.2K, in which case the measurement must be made while the sample is in a magnetic field greater than 4000G in order to assure that it is fully in the normal-conducting state and not in a superconducting state.

5.3 *Mechanical Measurements*

Measurements shall be performed in accordance with ASTM E-8 to determine Yield strength, Tensile strength and Elongation. Both longitudinal (to the rolling direction) and transverse (perpendicular to rolling direction) sections shall be examined as described in the following paragraph. For the tensile measurements, the rate of strain shall not exceed 1%/minute through the 0.2% offset yield strength. The rate of strain may be increased 5%/minute thereafter. HV has to be measured after final annealing for each sheet. The grain size shall be measured in accordance with ASTM E-112, Section 3.1.3.

For the surface finish, both sides of each sheet must pass a 1X visual inspection as follows: Inspections must be performed with the naked eye or correction to 20-20 vision, at a distance of no more than 10 inches and with the aid of a 100 watt light source. Magnifying aids may be used to determine the following causes for rejection:

- Pores, indentations or "orange peeling".
- Scratches deeper than .001 inch.
- Dirt or grease deposits.

Any of which are cause for whole part rejection. Corrective action may be proposed to FNAL for consideration. Minimum acceptable sheet finish may be determined by direct comparison of the sheet product and a FNAL supplied standard, and may be witnessed by a FNAL representative.

5.4 *Micrographs*

Photomicrographs shall be obtained as per ASTM Method E 883 as follows: Both through-thickness longitudinal (along rolling direction) and transverse (perpendicular to rolling direction) sections shall be prepared as per ASTM E3. One representative photomicrograph at x500 magnification per sample orientation (longitudinal and transverse) and one representative photomicrograph at x50 magnification per sample orientation shall be taken.

6.0 **QUALITY ASSURANCE**

The seller shall maintain a documented quality assurance program that will insure that each item offered for acceptance or approval conforms to the requirements

herein. A Quality Assurance plan must be submitted with the proposal and must be approved prior to the award of the contract.

6.1 *Documentation*

In particular, the seller will provide records of all inspections and tests. In addition, the following test data are required with the material delivery:

- Chemical analysis (ingot and final product); specific values per Section 3.1
- Residual resistivity ratio; specific values per Section 4.1.
- Mechanical properties; specific values per Section 4.4
- Grain size; specific values per Section 4.3
- Surface finish per Section 4.5
- Micrographs per Section 5.4

6.2 *Inspection*

FNAL reserves the right to have its technical or procurement representatives witness manufacturing steps, tests and inspections established under the seller's quality assurance program to demonstrate compliance with the specifications. Any information of a proprietary nature must be identified in the bid process.

7.0 **SHIPPING**

The material shall be coated on all sides by an adhesive PVC coating. Note that the PVC coating material should not contain any silicone. The material shall be individually wrapped or paper interleaved with non-abrasive, anti-tarnish paper to prevent surface damage. Wrapped sheets shall be packed for shipment in a way to prevent damage during normal hazards to shipment. The containers shall be constructed such that they can be handled by forklift and no palletized load shall exceed 2,500 pounds.

8.0 **ACCEPTANCE**

FNAL reserves the right to perform metallographic, chemical, mechanical, ultrasonic, eddy current and RRR tests on the delivered material. If the results of these tests show that the delivered material is not in compliance with sections 3.0 and 4.0 of this specifications, the material will be submitted to a third party testing laboratory which is acceptable to both FNAL and the vendor. Costs for such tests shall be shared between FNAL and the vendor. If portions of the material are not acceptable to FNAL, the vendor is obligated to provide replacement material.

All documentation as per 6.1 should be submitted to FNAL either prior to or with shipment. An identification system shall be carried with material to provide cross-reference to the test documentation.